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14. ABSTRACT

A used calibrated surface profilometer (Dektak 3) and a refurbished photoresist spin coater (CEE 200X) was purchased to supplement our U.S. Army Research Office (ARO) grant W911NF-12-2-0035, "Band-Offset Characterizations of Semiconductor Heterojunctions". These two instruments has met our immediate need to perform the etching and deposition of metal contacts for our detector samples in-house which helped us process the detector samples. The profilometer allowed us to precisely monitor the etched depth in the processed samples

15. SUBJECT TERMS

Profilometer, Depth analysis, device processing

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Report Title

Final Report: Equipment for Topographical Preparation and Analysis of Various Semiconductor Infrared Detector Samples

ABSTRACT

A used calibrated surface profilometer (Dektak 3) and a refurbished photoresist spin coater (CEE 200X) was purchased to supplement our U.S. Army Research Office (ARO) grant W911NF-12-2-0035, "Band-Offset Characterizations of Semiconductor Heterojunctions". These two instruments has met our immediate need to perform the etching and deposition of metal contacts for our detector samples in-house which helped us process the detector samples. The profilometer allowed us to precisely monitor the etched depth in the processed samples especially when we need to identify the issues related to unexpected impedance values of the devices. Although not supported by this grant, undergraduates, graduate students and postdocs used these instruments to learn processing steps related to device development.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received   Paper

      TOTAL:

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received   Paper

      TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Number of Presentations: 0.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

<u>Received</u> <u>Paper</u>

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

<u>Received</u> <u>Paper</u>

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

<u>Received</u> <u>Paper</u>

TOTAL:

Number of Manuscripts:

Books

Received   Book

TOTAL:

Received   Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

Graduate Students

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

NAME	PERCENT_SUPPORTED

FTE Equivalent:
Total Number:

Names of Under Graduate students supported

NAME	PERCENT_SUPPORTED

FTE Equivalent:
Total Number:

Student Metrics
This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:...... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):...... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:...... 0.00

Names of Personnel receiving masters degrees

NAME

Total Number:

Names of personnel receiving PHDs

NAME

Total Number:

Names of other research staff

NAME	PERCENT_SUPPORTED

FTE Equivalent:
Total Number:

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

A detailed description of the instrument with some examples of measuring the etching depth of the detector samples is presented in the attached document.

Technology Transfer

We have worked with Dr. P Wijewarnasuriya at the Army Research Lab to understand the bandd offsets of HgCdTe infrared detector structures. Especially when a sample is not performing as expected, the profilometer is used to measure the etch depth to rule out to confirm or to determine the etch depths.

Dektak 3 Profilometer: Operation and Results

Table of contents

Introduction

The Dektak 3 Surface Profilometer can be used to measure variations in thickness of surfaces. This instrument can also be used to verify the etch depth of the detector samples to make sure the device processing steps are as expected.

The Basic operation of the profilometer consists of a diamond stylus being moved in the x-y plane along the x-axis while making contact with the sample for a specified distance under a controllable contact force. This allows the instrument to measure small surface variations in z-axis stylus displacement as a function of its position along the x-axis. The Dektak profilometer can measure small vertical features on the sample ranging from 100 Å to 655 kÅ. The vertical resolution is ~10 Å.

The vertical movement of the stylus on the sample generates an analog signal which under goes an analog to digital conversion. This signal is stored analyzed and displayed on the computer interface.

The radius of the diamond stylus is 12.5 microns and the horizontal resolution is determined by the scan length and scan speed. There is a horizontal broadening factor which is a function of the radius of the stylus and the step height. This broadening factor is added to the horizontal dimensions of the steps via the software interface. The factory set tracking force of the stylus is 0.5 mN.

The video camera with variable magnification allows for manual placement of the stylus and the software allows to set the parameters for scan length and speed. Calibration of the data is done via software and can be saved for further analysis.

Instrument Specifications

- Vertical Range: 100Å to 655 KÅ
- Vertical Resolution: 10 Å
- Scan Length Range: 50 μm to 30 mm
- Scan Speed Ranges: Low, Medium, High (discussed further in instrument procedure)
- Scan Time Range: 3 seconds to 50 seconds
- Software Leveling: Two-point programmable or cursor leveling
- Stage Leveling: Manual
- Stylus (standard): Diamond, 12.5 μm radius
- Stylus Tracking Force: Adjustable, 0.1 mN to 0.5 mN
- Maximum Sample Thickness: 20mm
- Sample Stage Diameter: 127mm
- Sample Stage Translation: X Axis, ±10mm
  (From center)   Y Axis, +10mm, -70mm
- Sample Stage Rotation: 360° continuous
- Maximum Sample Weight: 0.5 kg
- Power Requirements: 100/115/200 V_{ac} ±10%, 50-60Hz, 200V_{ac}
- Warm-up Time: 15 minutes recommended for maximum
  Stability
- Operating Temperature: 21° C, ±3°C
  (70° F, ±5°F)
- Zoom Magnification: 90X (60-420X optional Video Zoom Optics)
- Camera: Solid state monochrome video image
- Sample Illumination: Variable intensity white light; IR & UV
  Blocked
- Dimensions: Scan Head 26cmW x 36cmD x 51cmH
  (10"W x 14"D x 20"H)

Measurement setup

Dektak 3 has an internal vibration isolation mechanism. Ambient temperature should be 21° C, ±3°C.

Figure 1. *The 90X solid state video camera with a variable intensity illuminator in the setup can be used to view the sample placed on the sample stage.*
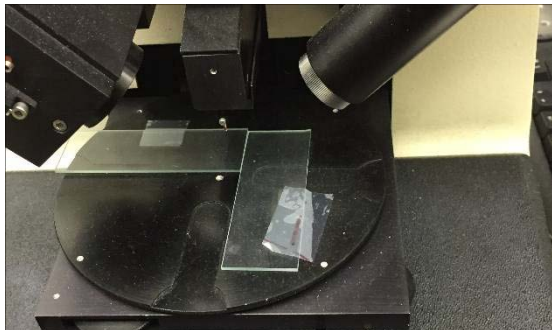

Figure 2. *The stage can be rotated 360 degrees. The sample stage is X-Y adjustable and accommodates samples up to 5 inches in diameter. The sample positioning is done manually on the stage guided by the image from the video camera.*

Figure 3. *The stylus that moves across the sample is circled in the picture. The Dektak 3 is a stylus based surface profiler. This means that the stylus is in physical contact with the sample. The radius of the standard stylus in our setup is 12.5 microns (as indicated by the red band around the stylus). This size meets most of the samples measurement requirements. However reducing the tip size necessitate the changing of the stylus force to a lower value.*

i. Scan speed and stylus force

When using a lower stylus force the stylus might lift off the surface for a large step at higher scan speeds.

For applications where a lighter stylus force required, it is recommended that the scans be done with a low or medium scan speed with the shortest possible scan length.

ii. Scan speed ranges

The Dektak 3 software specifies 3 scan speeds that can be chosen.

Speed	Resolution
Default Low (50 seconds)	High (2,000 data points per scan)
Default Medium (13 seconds)	Medium (500 data points per scan)
Default High (3 to 8 seconds)	Low (125 data points per scan)

Table 1: The scan speeds have three settings and the affect the resolution of the measurement

The scan length can be from 50 microns to 30 millimeters.

The following formula is used to find the number of data points for any given scan length and speed.

$$Number\ of\ Data\ points\ in\ scan = \frac{Scan\ length\ (in\ microns)}{Horizontal\ resolution\ (in\ microns)}$$

The horizontal resolution is directly related to the scan length and number of data points per scan. Therefore the number of data points per scan can be adjusted to the requirements of the measurement by altering the scan speed or length.

Scan speed – Indicates the time for a scan

Scan resolution – distance between data points (in microns per sample)

Measurement Procedure

i. Power on

The Dektak 3 and the computer are kept turned on with the illumination light for the stage turned off. The system is kept on because each time it is restarted the camera will reinitialize and the video settings have to be reset.
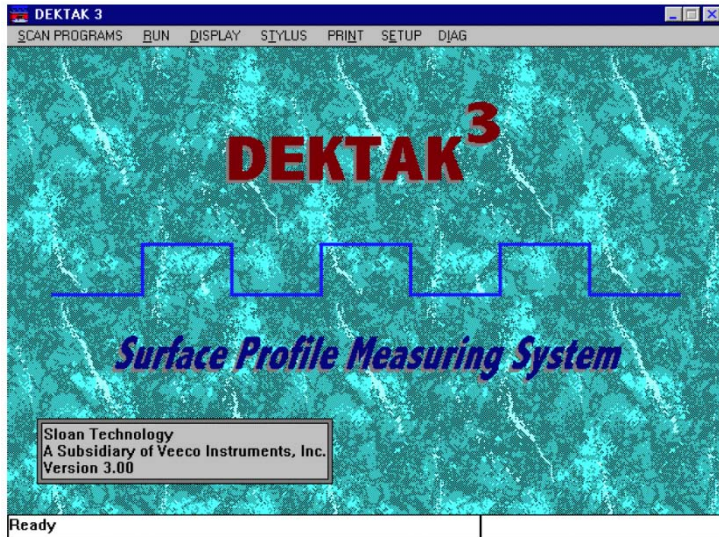
Figure 4. *This will open the both video overlay and the Dektak software at once. The video overlay is the feed from the video camera that is superimposed on the trace measured.*

ii. <u>Sample placement</u>

The wafer or processed sample should be placed on the sample stage fixed so that it does not move when the stylus is in contact.
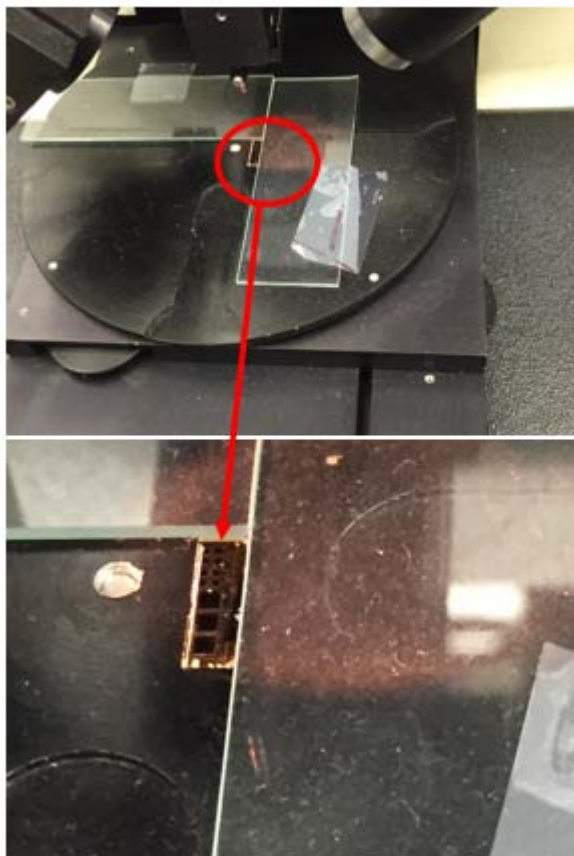

Figure 5. *The two glass slides hold the sample in place so it does not slide across the stage. Observation of the sample under the stylus is done with the eye and the video image. If the sample is not vertically aligned with the stylus the sample stage must be adjusted. Since the fine position adjustment is related to the video image the sample must have proper illumination and magnification prior to the actual positioning. To view the video image click "Display" and "Sample positioning" on software menu.*
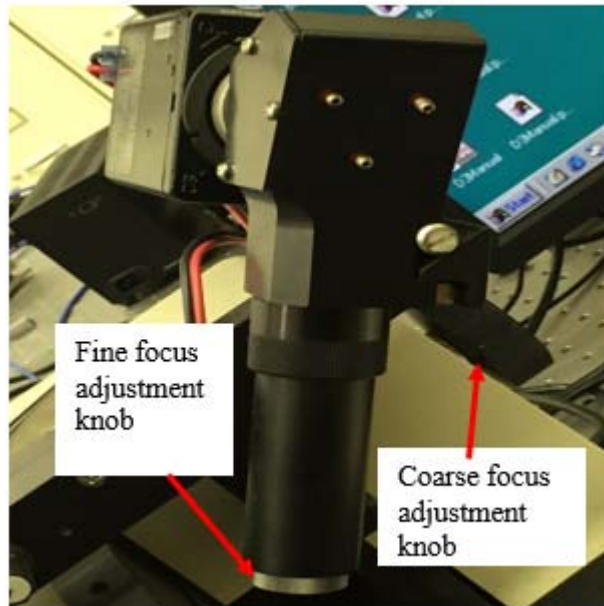
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iii. <u>Focus adjustment</u>

Figure 6. *Coarse focus adjustment is done by raising or lowering the optical tower using the large black focus knob on the right side of the scan head. Fine focus adjustment is done using the focus adjustment on the tip of the camera lens.*

iv.   <u>Illumination adjustment</u>

Figure 7. *Sample illumination is controlled by the illumination control knob. Turning it clockwise will increases the intensity of the light. Turning it completely counterclockwise will turn off the light.*

v. Stylus reticule alignment

a) To align reticule with the stylus tip vertically use the focus knob to raise or lower the optical stage.
b) Click "STYLUS" and "STYLUS DOWN"
c) To align the reticule with the tip horizontally select "SET UP" from the system menu and click on "STYLUS RETICULE". Out of the two options "ALIGN" and "RESET" select align and manually reposition the reticule.
d) Screen will show the stylus and its reflection as below.
e) Align the crosshair with the tip and double click. Click "YES" to update reticule position.
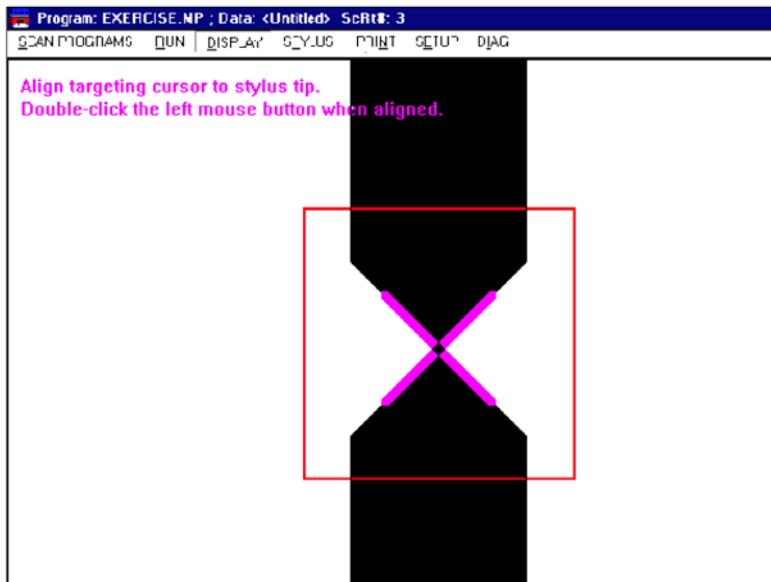

Figure 8. *The stylus reticule shows where the stylus will touch down when lowered. The camera zoom must be set to maximum when repositioning the reticule. The shadow of the tip on the sample will not be clearly visible if the sample is not properly illuminated. That will lead to incorrect alignment of the reticule. The illumination is adjusted so that the sharpest image is obtained.*
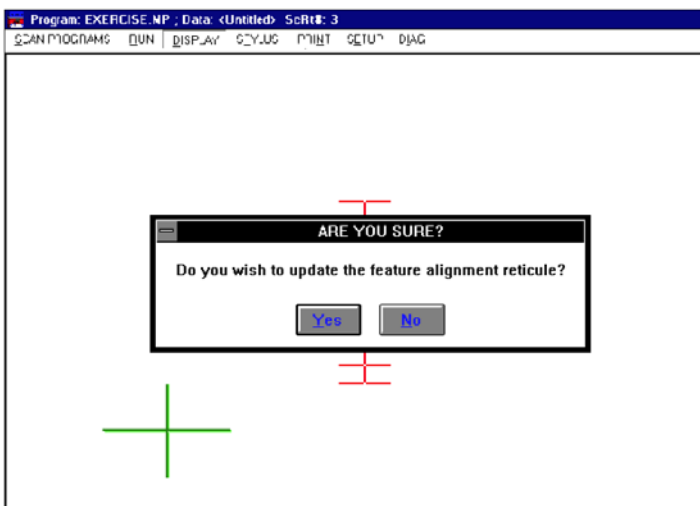
vi. Feature reticule alignment

a) Use the mouse to maneuver the reticule to the location on s*creen.*
b) *Once align*ed with feature double click the right mouse button.
c) Select "YES" for the realignment on the dialog box.

Figure 9. *The Feature reticule is a smaller green reticule displayed on the sample positioning stage. It can be aligned with a feature on the sample away from the stylus to more accurately position the stylus for scanning.*

vii. Lowering or raising the stylus

a) To raise the stylus off the surface click "STYLUS" and select stylus up.
b) It can also be raised using the F3 Key on the keyboard.

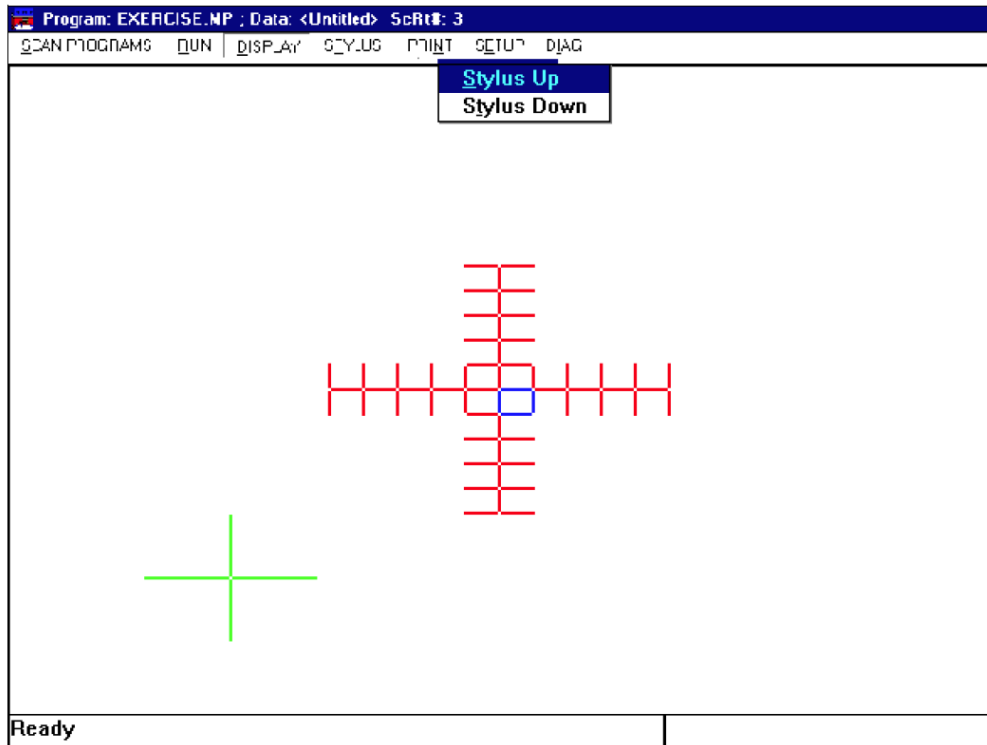Figure 10. *When repositioning the sample stage the stylus must be raised to ensure that the sample or stylus is not damaged.*

viii. <u>Sample stage positioning</u>

Once the above procedures have been completed the fine X-Y stage positioning can be done. This is done manually on the Dektak 3.

Sample positioning is done via turning the precision thumbwheels located on the front of the sample stage.

a) The wheel on the left side of the stage moves the stage along the X-axis (side to side) and the total range is 0.8 inches
b) The wheel on the right moves the stage along the Y-axis (in and out) with a total range of 3 inches.
c) Theta rotation is done by manually rotating the circular stage. 360° rotation is possible.
d) The large thumbwheel on the bottom of the stage is used to level the stage.

The video camera is mounted at a 90° to the stage. When the stage is translated side-to-side the video image on the monitor will move top-to-bottom. Likewise front-to-back stage translation will be displayed as side-to-side motion on the monitor.

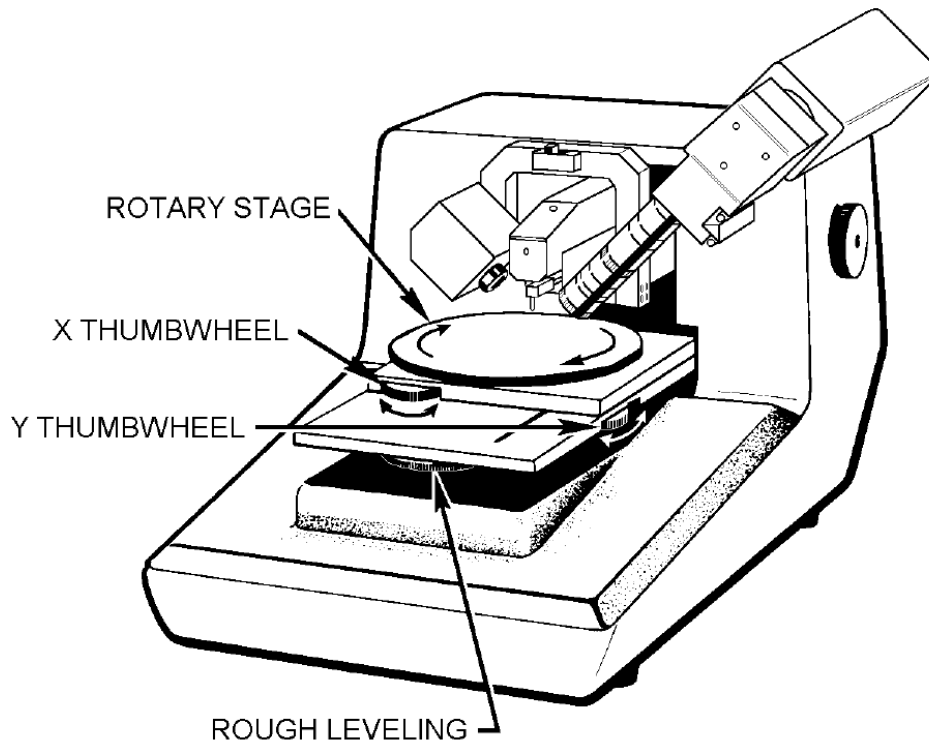Figure 11. *The figure shows the positions of the X and Y thumbwheels and the Rough leveling wheel.*

ix.   **Stylus positioning before starting a scan**
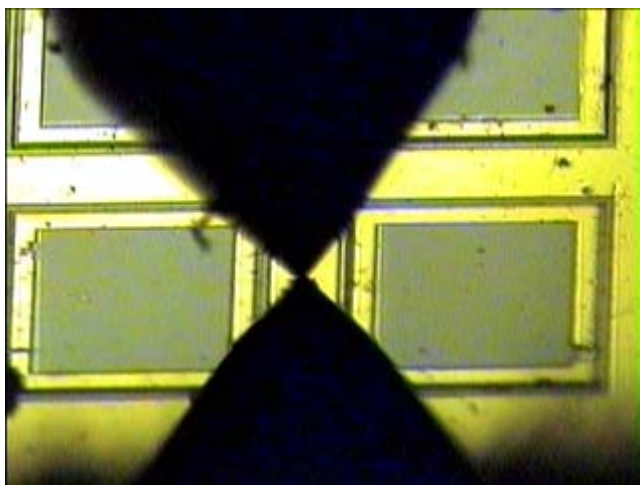

Figure 12. *The Stylus can be seen on surface just left of the sample to be measured.*

a) The stylus should be positioned just to the left of the feature to be measured as shown.

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b) During the scan the sample will move across the screen from right to left under the stationary stylus.

x. Single scan operation with software

a) Click on "SCAN PROGRAMS" from the system menu and select "Scan Routine".
b) Click on Scan length parameter the parameter window will be displayed.
c) Click on the "LENGTH" box and enter the value for the scan length.
d) Select the speed (Our samples (Low or Medium speed is best)
e) Select the "HILLS AND VALLEYS" profile.
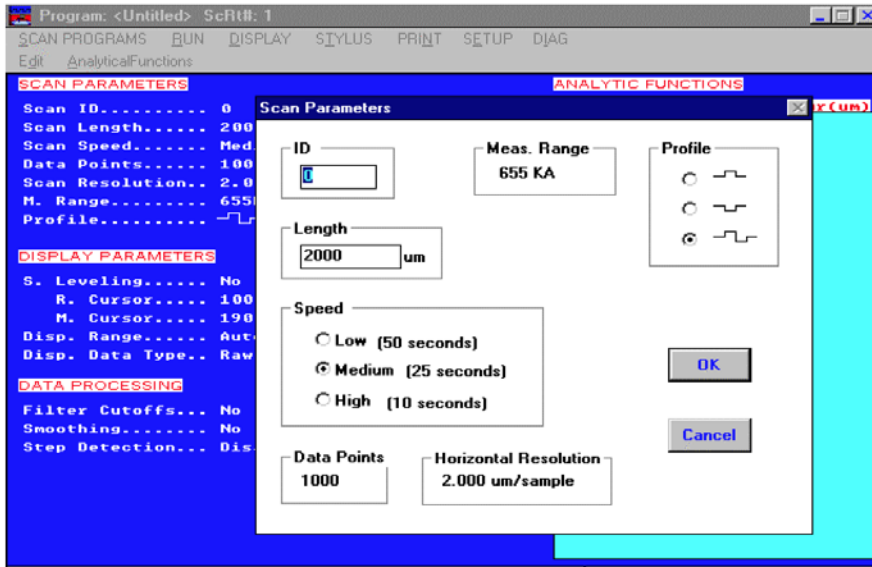
Figure 13. *With thee Scan Routine Window the scan length, the type of profile, and the scan speed can be changed.*

f) Click OK
g) Pull down the "RUN" menu and select "Run Single Scan"
h) The image of the sample will come on the screen super imposed on a scaled grid.
i) Stylus will lower onto the surface and sample will move from right to left and the trace will plot continuously.
j) Once scan is done the stylus will rise and the stage will return to original X-Y position.
k) The profile is recalculated and plotted on screen as below.
l)

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Figure 14. *This shows raw trace of a single step on a calibration standard before any software leveling.*
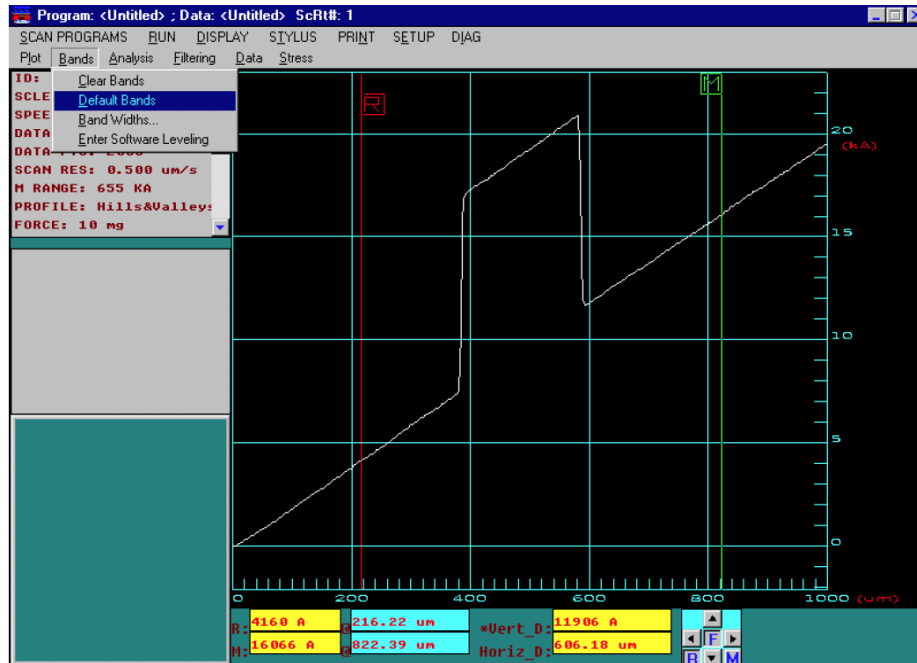
xi. Cursor Positioning

The Dektak 3 program contains two cursors, the reference (R) and measurement (M) which are used to define the portion of the trace to be used for leveling and performing analytical functions.

The cursor bandwidth can be adjusted for leveling and measurement.

I. Cursor positioning using mouse
   a) The left and right arrow boxes on the bottom of the screen can be used to position the cursors.
   b) Clicking the "R" and "M" buttons will select the reference and measurement cursors respectively.
   c) The up and down arrow boxes control the cursor bandwidth.
   d) Also clicking on the desired position on the grid will position the selected cursor at that location.

II. Cursor positioning using keyboard
   a) The blue boxes indicating the cursor locations can be manually changed to reposition the cursors.
   b) The arrow keys on the keyboard can be used to move the selected cursor.

xii. Manual Leveling

The stage must be manually leveled before doing the software leveling.

a) Stage leveling is done while a scan is in progress.
b) As the stage is moving and a trace is being generated on screen, the leveling thumb wheel is turned until the profile is tracking a horizontal line.
c) Clockwise rotation of the thumbwheel raises the stage and counterclockwise will lower the stage.
d) To verify the maximum possible level is obtained, the cursors should be placed to intersect the same horizontal plane on the trace.
e) Use the slope analytical function to determine what degree the stage is out of level. If the angle is greater than $\pm 0.01^{\circ}$ repeat above steps to get maximum possible level.

xiii. <u>Software leveling</u>

After being manually leveled software leveling will reduce the error further. Software leveling must be done to use analytical functions and to obtain accurate step height measurements.

a) Position R and M along the same horizontal plane far apart as possible. If the trace is rough cursor bandwidths should be increased to obtain best average.
b) Click on "Plot" and select "Level"
c) The trace will be replotted with R and M cursors intercepts at zero.

xiv. <u>Setting Zero point</u>

Any point on the trace may be selected as zero point. The zero point will be the point of reference for all measurements taken. When you set zero point it selects R cursor intercept as zero point.

xv. <u>Step Height measurement</u>

Once the trace has been properly leveled and zeroed an accurate step height measurement can be done.

a) M Cursor should be positioned at the top of a step at a point that is smooth and free of roughness.
b) R Cursor should be positioned at the bottom of the step.
c) The value of the step height is displayed in the box labelled "Vert. D:"
d) To average the data points within cursor bandwidths the Average Step Height analytical function is used.

xvi. <u>Analytical Function usage</u>

The Dektak 3 has many useful analytical functions one of which is the Average Step Height function. It calculates the vertical difference between the average bandwidths for M and R cursors.

a) Click on Analytical Functions on the menu bar and select Delta Average Step Height function (ASH) and click "COMPUTE".

b) Select the bandwidths as shown below and Select "Measure and Program" to enter it into the scan routine. This will ensure that the ASH is calculated each time the routine is run.
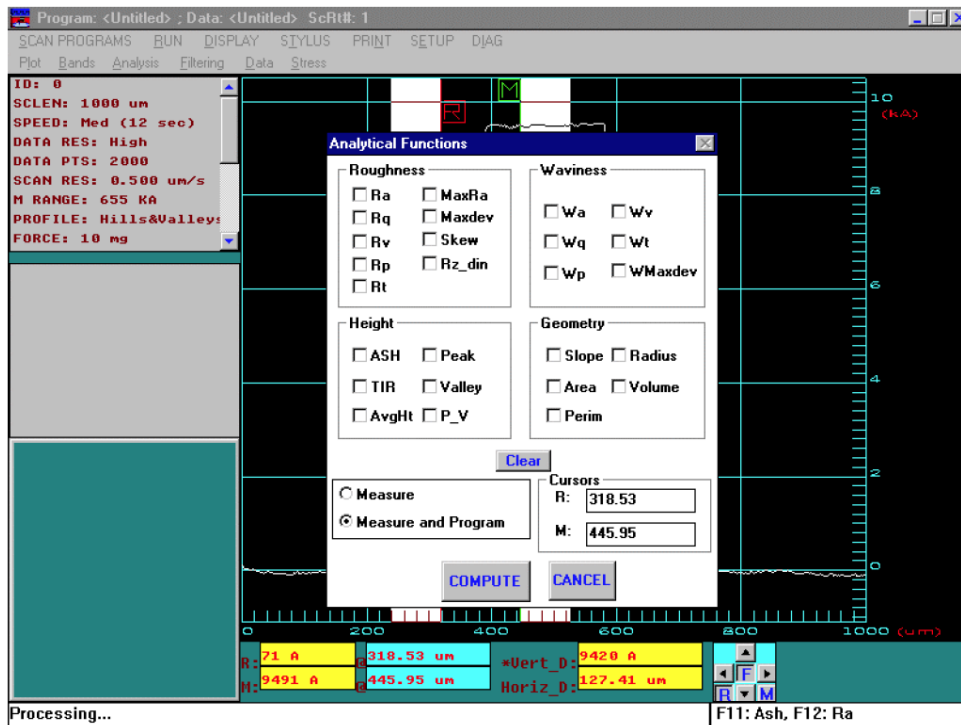

Figure 15. *This shows the different analytical functions available on the Dektak software. The most important categories are the Height and Geometry functions.*

xvii. <u>Analytical functions</u>

The Dektak 3 software has 26 different analytical functions. A brief description of each function that is useful to us is given below.

Step Height Parameters
When calculating step height parameters, the R and M cursors are used to define the assessment area of the profile trace. The stage should be leveled and the scan trace should be software leveled prior to calculating any analytical function.

ASH: Used to obtain a step height measurement in applications where roughness or noise is present on the profile trace. It computes the difference between two Average height measurements.

AVG HT: Calculates the average height of a step, with respect to the zero line, using the R and M cursors to define the area of measurement.

PEAK: Calculates the maximum height above the baseline, as determined by the cursor/trace intercepts.

PV: Calculates the vertical distance between the maximum peak and maximum valley.

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TIR: Calculates the vertical distance between the highest and lowest data points between the cursors.

VALLEY: Calculates the maximum depth below the baseline, determined by the cursor/trace intercepts.

Geometry Parameters
When calculating step height parameters, the R and M cursors are used to define the assessment area of the profile trace. The stage should be leveled and the scan trace should be software leveled prior to calculating any analytical function.

Area: Computes the area of a profile between the R and M cursors with respect to the horizontal zero grid line. The profile **must be leveled** for accurate results. If the profile is above the zero line, area is expressed as a positive value in square microns. If the profile is below the zero line, the result will be a negative value.

Perim: Calculates the outside perimeter of a profile between the R and M cursors. A horizontal reference line is created using the R and M cursor intercepts. The profile must be leveled for accurate results.

Radius: A least-squares-arc is fitted to the data points and the radius is calculated from the equation of a circle. The algorithm does not distinguish between concave and convex shapes. To maximize the accuracy of the results, the following factors must be considered: (1) the sample shape must approximate a sector of a circle: (2) the stylus tip must traverse the apex of the sample if it is a sphere. Using the largest radius stylus possible will help minimize the error; (3) Repeatability errors may dominate the measurement if the chord rise is less than 100Å for scans longer than 1mm.

Slope: Calculates the arc tangent of the ratio of the vertical distance to the horizontal distance between the R and M cursor/trace intercepts. The result is expressed in degrees. Slope is
Useful only for relatively shallow slopes. If the stylus radius is too large or the step too steep, the stylus will contact the upper edge of the step before the lower edge and the slope measurement will be inaccurate.

Volume: The integration-by-shells technique is used to find the volume of a solid. This is accomplished by rotating the lamina delineated by the scan trace and a line segment connecting the cursor intercepts through 180 degrees about a vertical axis which is located half way between the cursors.

Measurements on detector structures

The Dektak 3 was used to measure and verify the thickness on a set of GaAs/Al$_x$Ga$_{1-x}$As quantum dot infrared detectors (G15-038 & G15-039) indicated below. The samples were also measured by the group at University of Leeds who carried out the processing of the sample. The Leeds group measurements were done on a RKLA Tencor P-15 Profilometer.

RKLA Tencor P-15 Profilometer (Profilometer at The University of LEEDS)

Roughness can be measured with up to a 0.5 Å resolution.

It has a maximum scan area of 200 X 200 mm.

Microscopic and Macroscopic Feature Resolution: Combines macroscopic and microscopic surface analysis, and measures features as small as 0.25 μm.

Sample	Design parameters (μm)		
	Total Thickness	**Top contact thickness**	**Bottom contact thickness**
G15-038A	1.706	0.2	0.8
G15-038B	1.706	0.2	0.8
G15-039B	1.666	0.2	0.8
G15-039C	1.666	0.2	0.8

Table 1: *The design parameters for the quantum dot detector structures are shown. There are two processed samples of each detector (G15-038 and G15-039).*

The four samples (2 each from each design) were processed with different etching depths for the Top contact and the bottom contact. To get to the bottom contact the samples have been etched down from the top contact to part of the bottom contact as indicated in Figure 16. The etching depth data were provided by the Leeds group who processed the sample. Those data are compared with the Dektak 3 data for the same samples.
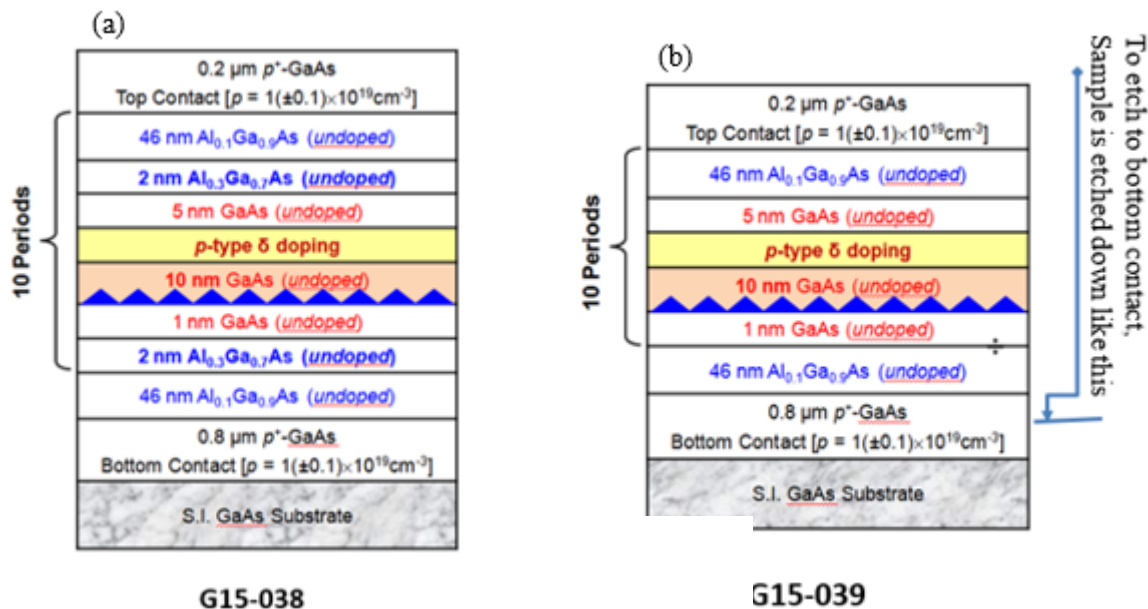
Device Structure

G15-038

G15-039

Figure 16. (a) is G15-038 and (b) is G15-039. *The two samples are designed with a total thickness of 1.706 μm for G15-038 and a total thickness of 1.666 μm for G15-39. The top and bottom contacts are the 0.2 μm p-doped GaAs layer and the 0.8 μm p-doped GaAs layer respectively. The top contact has a window etched into it for incident radiation. The samples has been etched from top to bottom to allow access to the bottom contact.*

The measurements done by the Dektak 3 on G15-038A and G15-038B samples is given below with a comparison with he Leeds group data.

Sample	Top etching(μm)		Bottom etching(μm)		Metal thickness (μm)	
	Leeds Data	Dektak profilometer data	Leeds Data	Dektak profilometer data	Leeds Data	Dektak profilometer data
G15-038A	0.160	0.154 ± 0.01	0.152-0.178	0.182 ± 0.01	0.189	0.1901 ± 0.01
G15-038B	0.176	0.186 ± 0.01	0.201-0.232	0.189 ± 0.01	0.198	0.1891 ± 0.01

Table 2: *The data taken with the Dektak 3 compared with the data provided by the Leeds group for the samples G15-038A and G15-038B.*
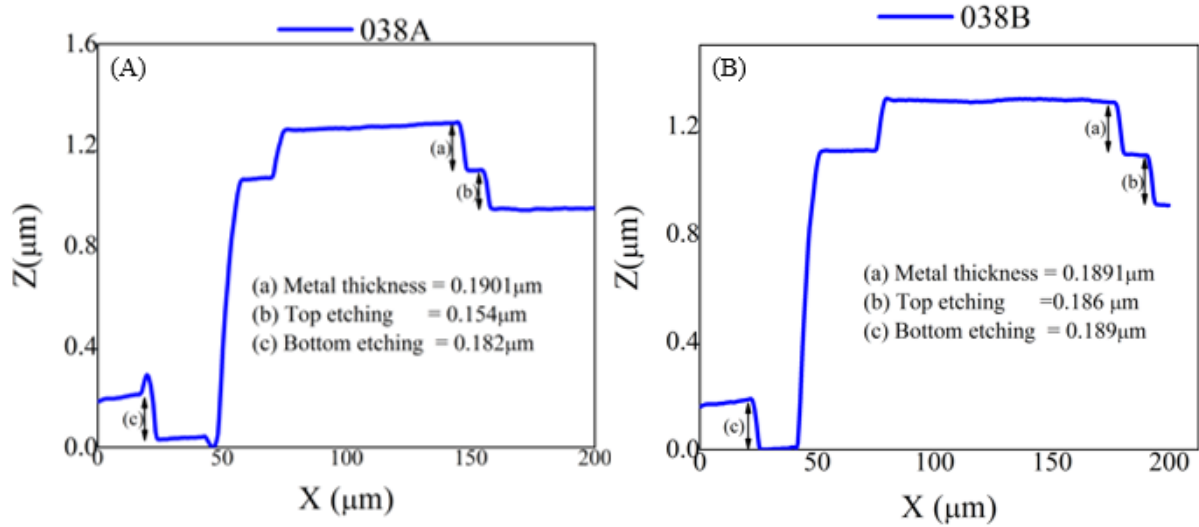

Figure 17. *(A) is the thickness profile of G15-038A and (B) is the thickness profile of G15-038B. In these figures the top most layer (a) is the gold metallization. And the second layer (b) is the p-doped GaAs top contact. The structures have been etched down to the bottom layer (c) to get access to the p-doped bottom contact.*

The data for the samples G15-039B and G15-039C are given below. The difference between these samples and the previous samples is that, these contain 2 additional undoped $Al_{0.3}Ga_{0.7}As$ layers increasing their overall thickness.

Sample	Top etching(μm)		Bottom etching(μm)		Metal thickness (μm)	
	Leeds Data	Dektak profilometer data	Leeds Data	Dektak profilometer data	Leeds Data	Dektak profilometer data
G15-039B	0.196	0.188 ± 0.01	0.144-0.150	0.165 ± 0.01	0.446	0.446 ± 0.01
G15-039C	0.198	0.194 ± 0.01	0.170-0.2409	0.184 ± 0.01	0.279	0.283 ± 0.01

Table 3: *The data taken with the Dektak 3 compared with the data provided by the Leeds group for the samples G15-039B and G15-039C.*
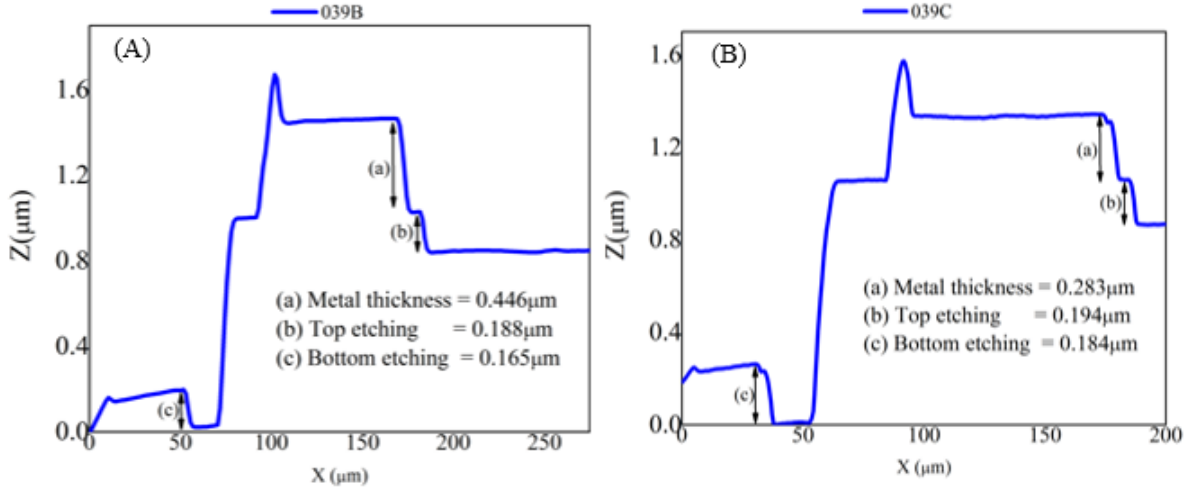

Figure 18. *(A) is the thickness profile of G15-039B (B) is the thickness profile of G15-039C. In these figures the top most layer (a) is the gold metallization. And the second layer (b) is the p-doped GaAs top contact. The structures have been etched down to the bottom layer (c) to get access to the p-doped bottom contact. The metal thickness is greater than the previous samples because metallization had to be done twice for these samples. That is clearly indicated here.*

The above data is the average of 5 scans for each sample and the measurements for each sample was taken at the same spot near to the center mesa in each sample. The difference of thickness is within the boundary limits of the measurements done by the Leeds group thereby verifying the growth parameters.

GSU13-MPD-GB1 Heterostructure

Also measurements were done on a GaAs/Al$_x$Ga$_{1-x}$As heterostructure on two different mesa sizes to verify their thickness and to check the calibration of the instrument.
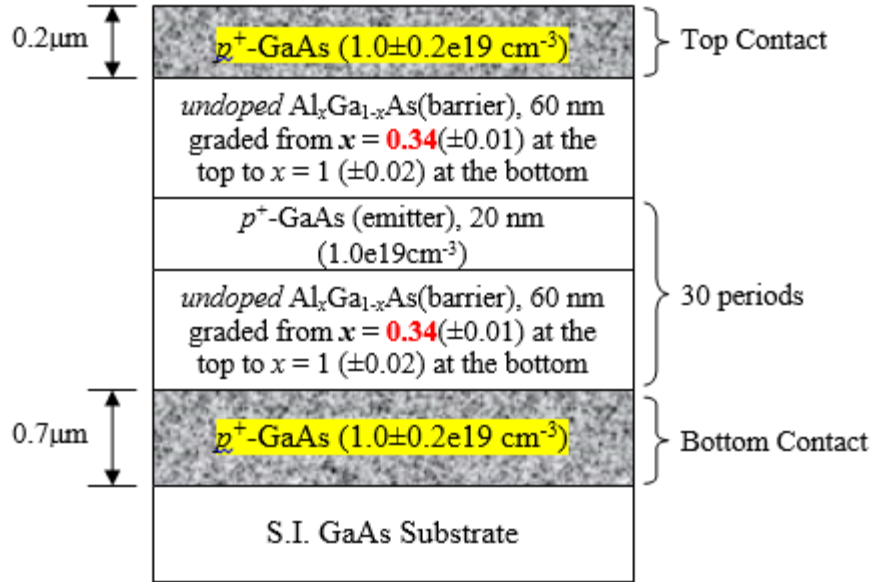

Figure 19. *GSU13-MPD-GB1 a multi-period heterostructure and the top and bottom contacts have been etched and metallized so that the wire bonding can be done. The total thickness of the design is 3.36 µm and the width of the metal contact is 70 µm.*
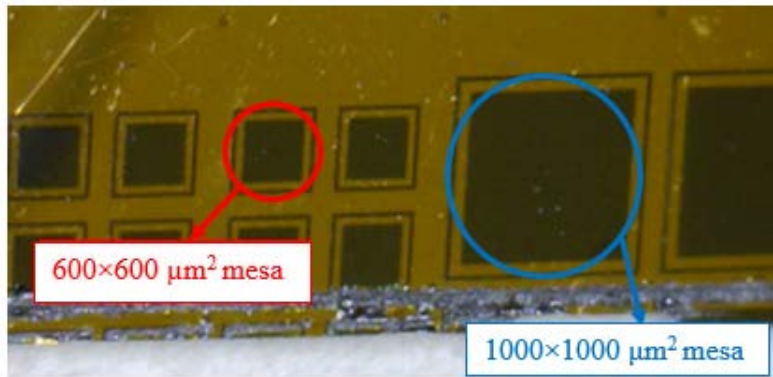

Figure 20. *The 600×600 µm^2 mesa circled in red and the 1000×1000 µm^2 mesa circled in blue were measured to verify the thickness and width of the top metal contacts and to check the calibration of the profilomter.*

The samples were measured with 800 µm scan length for the smaller mesa and 2000 µm scan length for the larger mesa. The waviness in the profiles are due to these larger scan lengths. The table below compares the design values and the values measured with the Dektak 3 profilometer.

| GSU13-MPD-GB1 | Design parameters (µm) | Our profilometer data (µm) | |
		600×600 µm^2 mesa	1000×1000 µm^2 mesa
Total thickness	3.36	3.602 ± 0.01	3.46 ± 0.01
Contact width	70	67.5 ± 0.01	70 ± 0.01

Table 4*: The data taken with Dektak profilometer compared with the design parameters.*

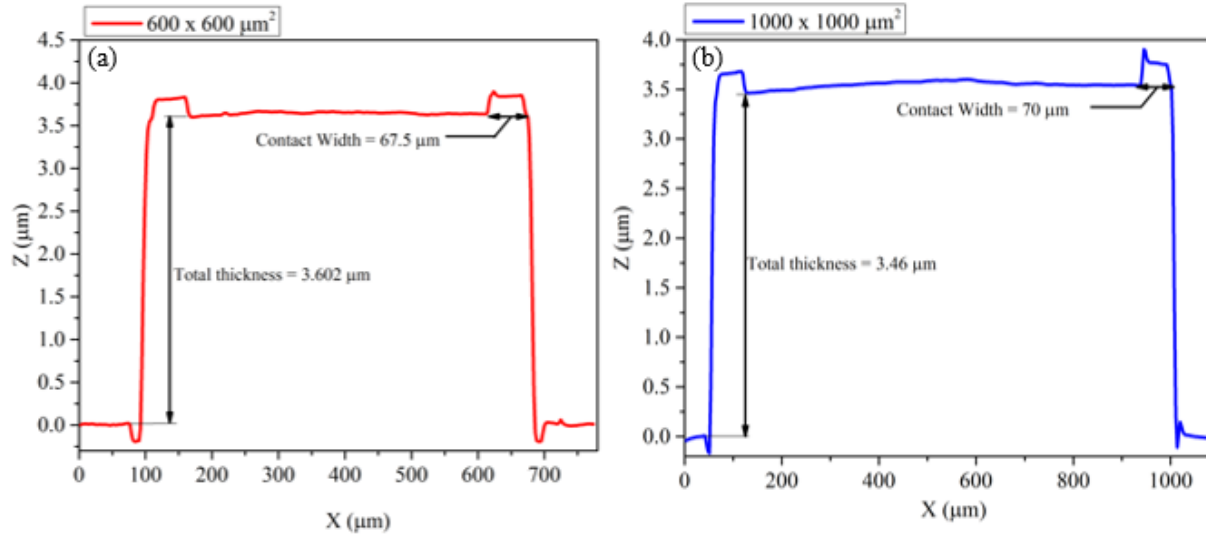**Figure 21.** *Thickness profile of two mesa (600×600 µm^2 and 1000×1000 µm^2) of the GSU13-MPD-GB1 sample. (a) is the thickness profile of the 600×600 µm^2 mesa and (b) is the thickness profile of the 1000×1000 µm^2 mesa. This measurement allowed us to verify the levelling and calibration of the instrument.*